



IN THE UNITED STATES PATENT & TRADEMARK OFFICE

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In re Application of Grosch et al.

Serial Number 09/324,271

Filed: June 2, 1999

For: SUSPENSIONS OF MULTIMETAL CYANIDE COMPOUND, THEIR PREPARATION AND USE

DECLARATION UNDER 37 CFR 1.132

I, Dr. Kathrin Harre, a citizen of the Federal Republic of Germany and residing at Dresden declare as follows:

I am a fully trained chemist, having studied chemistry at the Universities of Halle and Mainz, from 1988 to 1993,

I was awarded my doctor's degree by the University of Mainz in 1997,

I am well acquainted with technical English,

I joined BASF Schwarzheide of 01986 Ludwigshafen, Federal Republic of Germany, in 1998, since when I have been working in the field of polyurethane research and development,

I am one of the inventors of the subject matter disclosed and claimed in U.S. Patent Application Serial Number. 09/324,271

In order to compare the catalytic activity of a catalyst suspension according to the above patent application compared with the activity of a wet filter cake and a dried catalyst the following experiments were performed.

Example 1 (catalyst preparation)

192 kg of an aqueous solution of hexacyanocobalt acid with a cobalt content of 9 g/l were added to a 800 L reactor equipped with a plate-like stirrer, a tube for dosing, and a pH electrode. The solution in the reactor was heated to 50°C while stirring. To this solution were added 108,7 kg of an aqueous solution of zinc acetate with a zinc content of 2.7 percent by weight which had been preheated to 50°C with continuous stirring by introducing a stirring power of 1W/l over a period of 45 minutes. 5,5 kg of an alkylene oxide block copolymer (Pluronic® PE 6200 of BASF, AG) was then added to this suspension while stirring. This was followed by the addition of 5,5 kg water and 41.1 kg of an aqueous solution of zinc acetate with a zinc content of 2.6 percent by weight over a period of 10 minutes which had been preheated to 50°C with continuous stirring by introducing a stirring power of 1W/l. The suspension was then heated to 55°C until the pH value dropped from 4.7 to 3.4 and remained constant at 3.4. The precipitate was then filtered and then washed with a 400 l water (example 1a). One portion of the wet filter cake was then dispersed in propoxylated glycerin with a molecular weight of 900 using a Mill (Fryma MZ 80). This suspension had a content of DMC catalyst of 6,2 percent by weight (example 1b). Another portion of the wet filter cake was vacuum dried over night at 25 mbar, 50°C, (example 1c).

Example 2

All catalysts were tested using a standard screening experiment. The catalyst in the amount of 25 ppm corresponding to the catalyst amount in the final product was mixed into 128 g of a polypropylene glycol with the molecular weight of 400 g/mol. To ensure an effective mixing and to avoid inhomogeneous catalyst distributions, a turrax mixer was used. The mixture was heated to 100°C. After a standard procedure of 2 h vacuum stripping to remove residual water and oxygen from the reaction mixture, the temperature was increased to 130 °C, and 72 g of propylene oxide were added to the reaction mixture within 2 min. Temperature and pressure were recorded. A sudden pressure and temperature change indicated the initiation of reaction. The induction times were criteria for the catalytic activity of the catalyst. The different induction times are summarized in table 1.

catalyst	Description	induction time [min]
example 1a	wet filter cake	20
example 1b	suspension	8
example 1c	dried catalyst	10

The examples show clearly the differences between the catalytic activities of the catalyst treated in different ways after precipitation. The catalyst suspension according to the current patent application clearly shows the highest activity.

I further declare that all statements made herein of my own knowledge are true and that all statements on information or belief are believed to be true; and further that these statements are made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code

and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at 01986 Schwarzheide, Germany, this 22 day of November 2001

A handwritten signature in black ink, appearing to read 'Kathrin', followed by a long horizontal flourish.

Signature of Declarant